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# 2,6-Diethylanilinium perchlorate

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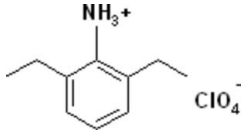
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.006 Å; disorder in main residue; $R$ factor = 0.089; $wR$ factor = 0.278; data-to-parameter ratio = 39.3.

The asymmetric unit of the title molecular salt, $C_{10}H_{16}N^+\cdot ClO_4^-$, contains two cations and two anions. The atoms of one of the ethyl side chains of one of the cations are disordered over two sets of sites in a 0.531 (13):0.469 (13) ratio. In the crystal, the components are linked by N—H···O and bifurcated N—H···(O,O) hydrogen bonds and weaker C—H···O interactions, such that the organic cations alternate with the perchlorate anions, forming ribbons in the *a*-axis direction.

## Related literature

For background to the physical properties and potential applications of molecular salts, see: Czarnecki *et al.* (1994); Mylrajan & Srinivasan (1991); Toumi Akriche *et al.* (2010); Xiao *et al.* (2005). For the graph-set notation of hydrogen-bond networks, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{10}H_{16}N^+\cdot ClO_4^-$
$M_r$ = 249.69
Monoclinic, $P2_1/c$
$a$ = 15.105 (3) Å
$b$ = 21.192 (5) Å
$c$ = 7.718 (6) Å
$\beta$ = 98.10 (3)°
$V$ = 2446 (2) Å$^3$
$Z$ = 8
Ag $K\alpha$ radiation
$\lambda$ = 0.56085 Å
$\mu$ = 0.17 mm$^{-1}$
$T$ = 293 K
0.50 × 0.40 × 0.20 mm

### Data collection

Enraf–Nonius TurboCAD-4 diffractometer
15750 measured reflections
11941 independent reflections
2954 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.052
2 standard reflections every 120 min
intensity decay: 5%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.089
$wR(F^2)$ = 0.278
$S$ = 0.91
11941 reflections
304 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.36 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.36 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$A$···O6 | 0.89 | 2.41 | 3.029 (4) | 127 |
| N1—H1$B$···O4 | 0.89 | 2.27 | 3.043 (5) | 146 |
| N1—H1$B$···O2$^{i}$ | 0.89 | 2.48 | 2.889 (4) | 109 |
| N1—H1$C$···O3$^{ii}$ | 0.89 | 2.10 | 2.935 (4) | 157 |
| N1—H1$C$···O2$^{i}$ | 0.89 | 2.58 | 2.889 (4) | 101 |
| N2—H2$A$···O1 | 0.89 | 2.17 | 2.971 (4) | 149 |
| N2—H2$B$···O5 | 0.89 | 2.39 | 2.875 (4) | 114 |
| N2—H2$B$···O7$^{iii}$ | 0.89 | 2.24 | 2.991 (4) | 141 |
| N2—H2$C$···O7$^{iv}$ | 0.89 | 2.03 | 2.805 (3) | 144 |
| C3—H3···O3$^{v}$ | 0.93 | 2.60 | 3.321 (5) | 134 |
| C13—H13···O8$^{vi}$ | 0.93 | 2.49 | 3.418 (6) | 172 |

Symmetry codes: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x, y, z-1$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $x, y, z+1$; (v) $-x, -y+1, -z+1$; (vi) $-x+1, -y+1, -z+1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5332).

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# supporting information

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## 2,6-Diethylanilinium perchlorate

**Wajda Smirani Sta, Mohamed Rzaigui and Salem S. Al-Deyab**

### S1. Comment

A special attention is focused on the synthesis of hybrid class of inorganic-organic materials because of their interesting architectures and wide variety of physical properties. Organic substructure of these compounds is usually responsible for their molecular hyperpolarizability, electric permittivity and spontaneous polarization. The inorganic part determines thermal and mechanical stability of the crystals. The combination of these various features attributed to both organic and inorganic substructures may lead to interesting materials (Xiao *et al.*, 2005). In particular, the association of the perchlorate anions to organic molecules could lead to materials having phase transitions, non-symmetric structures, characteristic H-bonds (Czarnecki *et al.*, 1994; Mylrajan & Srinivasan, 1991). Among the crystals comprising perchlorate anions, the most interesting are non-centrosymmetric, due to non linear optical(ONL) properties. A pronounced second harmonic generation was found in L-leucinium perchlorate (SHG efficiency d eff= 0.44 x d eff KDP). In this paper, we report single-crystal X-ray study of 2,6-diethylanilinium perchlorate (I). Crystal structure of this latter is depicted in the figure 1. The asymmetric unit, built of two 2,6-diethylanilinium cations and two perchlorate anions, has the geometrical configuration shown in the figure 2. These four components establish between them H-bonds to form a tetra-membered ring. This ring form two slightly corrugated ribbons parallel to the a direction at y = 1/4 and 3/4. Each ribbon is built of an alternance of both inorganic and organic entities.

The first $Cl(1)O_4^-$ is surrounded by three $N(1)H_3^+$cations, to build a ribbon extended in the a direction, and generating $R_2^4(8)$ graph-set motifs.Whereas the second $Cl(2)O_4^-$, surrounded by two $N(2)H_3^+$cations, leads to $R_2^4(10)$ graph-set motifs which form another ribbon extended in the same direction. Both parallel ribbons are attached together by N—H···O hydrogen bonds. The organic molecules are anchored on these ribbons so that to leave spacious channels (13.9 Å x 3.3 Å) parallel to the a axis. The Cl—O distances indicate rather slight distortion of the two perchlorate anions from the tetrahedral symmetry. The shortest Cl—O bond equals to 1.384 (3) Å, whereas the longest one to 1.444 (3) Å, the angles vary from 105.6 (2)° to 111.2 (2)° that are standard values for perchlorate ions (Toumi Akriche, S. *et al.* 2010). In this organisation, the components display different interactions (electrostatic, H-bonds, Van derWalls) to keep the three-dimensional network stability.

### S2. Experimental

An ethalonic 2,6-diethylaniline solution (5 mmol, in 5 ml) was added to an aqueous perchloric acid solution (0.5 M, 10 ml) at room temperature (293 K). Slow evaporation of the obtained mixture led to the formation of small crystals of the title compound. These were recrystallised from a mixture of water / ethanol (80% / 20%) to yield colourless blocks of (I).

### S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97Å, N—H = 0.89Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(carrier) or 1.5$U_{eq}$(methyl C).

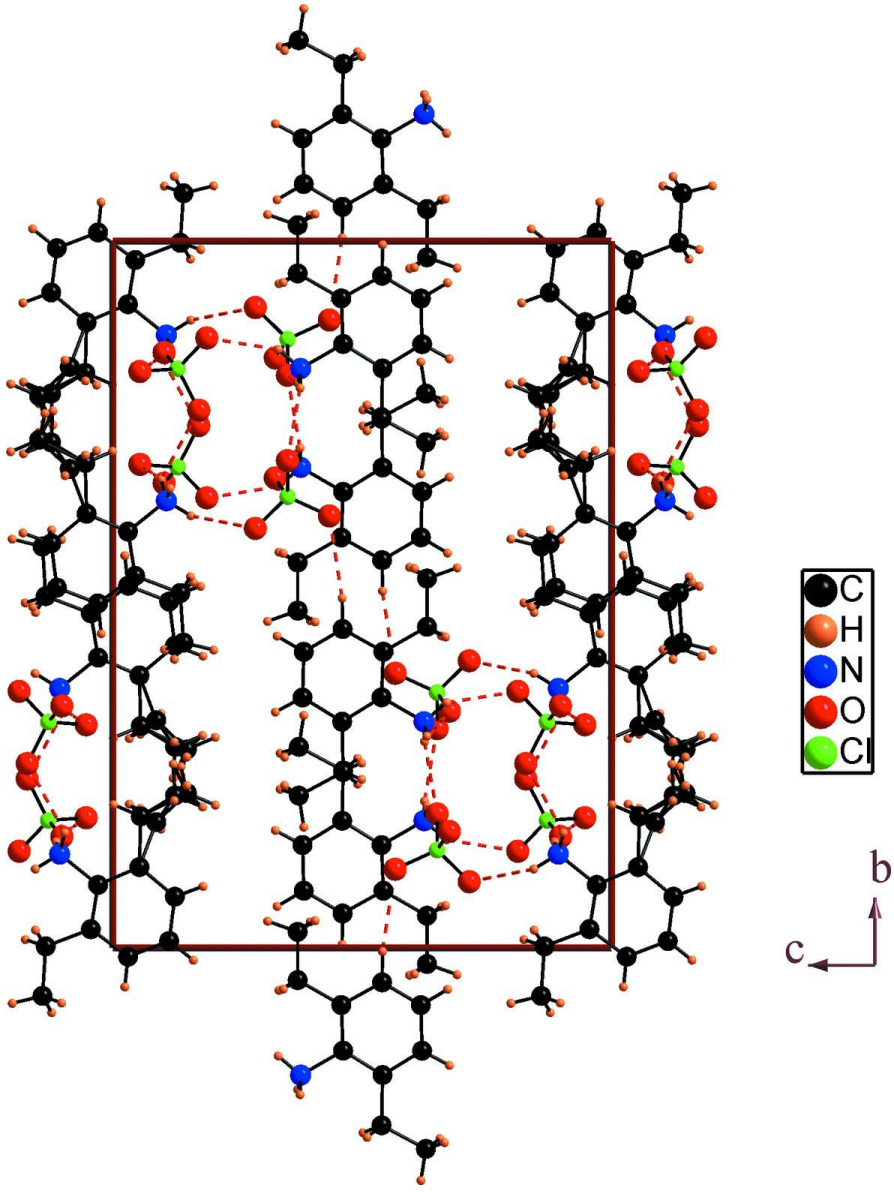


**Figure 1**

A view of the packing of (I) along the a axis.

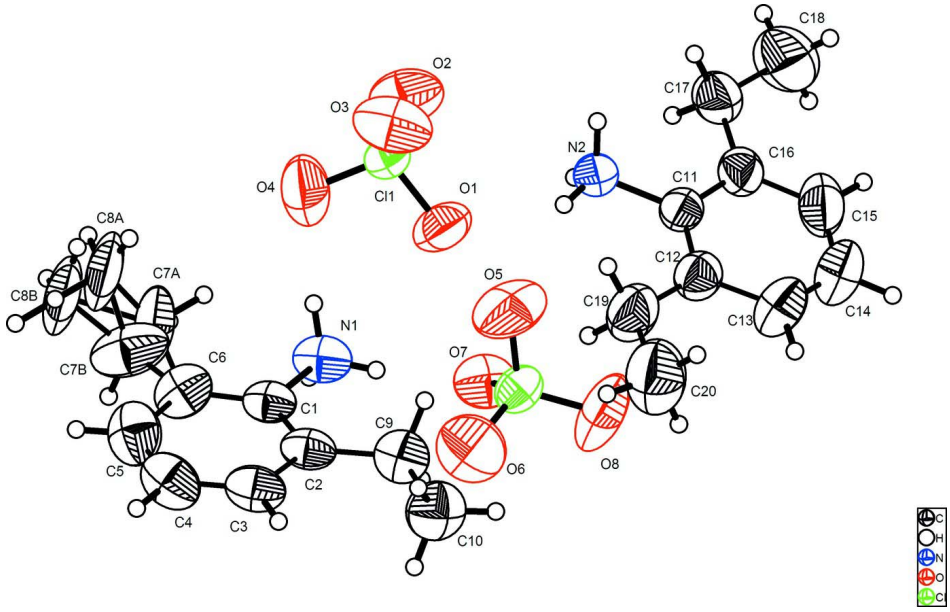


**Figure 2**

The molecular structure of (I) with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

### 2,6-Diethylanilinium perchlorate

*Crystal data*

$C_{10}H_{16}N^{+}\cdot ClO_4^{-}$
$M_r = 249.69$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.105\ (3)$ Å
$b = 21.192\ (5)$ Å
$c = 7.718\ (6)$ Å
$\beta = 98.10\ (3)°$
$V = 2446\ (2)$ Å$^3$
$Z = 8$
$F(000) = 1056$
$D_x = 1.356$ Mg m$^{-3}$
Ag $K\alpha$ radiation, $\lambda = 0.56085$ Å
Cell parameters from 25 reflections
$\theta = 9–11°$
$\mu = 0.17$ mm$^{-1}$
$T = 293$ K
Block, colourless
0.50 × 0.40 × 0.20 mm

*Data collection*

Enraf–Nonius TurboCAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled $\omega$ scans
15750 measured reflections
11941 independent reflections
2954 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 28.0°$, $\theta_{min} = 2.2°$
$h = -25\rightarrow5$
$k = -35\rightarrow0$
$l = -12\rightarrow12$
2 standard reflections every 120 min
intensity decay: 5%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.089$
$wR(F^2) = 0.278$
$S = 0.91$
11941 reflections
304 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.1095P)^2 + ]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.36$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor wR and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) etc. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C7A | −0.0545 (10) | 0.6898 (8) | 0.139 (3) | 0.094 (7) | 0.469 (13) |
| H7A1 | 0.0024 | 0.7060 | 0.1960 | 0.112* | 0.469 (13) |
| H7A2 | −0.0600 | 0.7012 | 0.0165 | 0.112* | 0.469 (13) |
| C7B | −0.0813 (12) | 0.6774 (8) | 0.089 (2) | 0.106 (6) | 0.531 (13) |
| H7B1 | −0.0280 | 0.7030 | 0.1180 | 0.127* | 0.531 (13) |
| H7B2 | −0.0885 | 0.6726 | −0.0373 | 0.127* | 0.531 (13) |
| C8A | −0.1146 (10) | 0.7157 (9) | 0.204 (3) | 0.126 (4) | 0.469 (13) |
| H8A1 | −0.1124 | 0.7604 | 0.1839 | 0.188* | 0.469 (13) |
| H8A2 | −0.1072 | 0.7076 | 0.3273 | 0.188* | 0.469 (13) |
| H8A3 | −0.1713 | 0.6995 | 0.1504 | 0.188* | 0.469 (13) |
| C8B | −0.1498 (9) | 0.7177 (8) | 0.116 (3) | 0.126 (4) | 0.531 (13) |
| H8B1 | −0.1429 | 0.7570 | 0.0571 | 0.188* | 0.531 (13) |
| H8B2 | −0.1481 | 0.7251 | 0.2387 | 0.188* | 0.531 (13) |
| H8B3 | −0.2061 | 0.6990 | 0.0695 | 0.188* | 0.531 (13) |
| N2 | 0.37636 (18) | 0.68014 (13) | 0.7366 (3) | 0.0541 (7) | |
| H2A | 0.3312 | 0.6531 | 0.7121 | 0.081* | |
| H2B | 0.3754 | 0.7078 | 0.6497 | 0.081* | |
| H2C | 0.3709 | 0.7005 | 0.8355 | 0.081* | |
| O3 | 0.1026 (2) | 0.65755 (15) | 0.8694 (4) | 0.1012 (11) | |
| O1 | 0.18825 (19) | 0.63681 (13) | 0.6498 (4) | 0.0866 (9) | |
| O7 | 0.3499 (2) | 0.68975 (12) | 0.0880 (3) | 0.0791 (8) | |
| N1 | 0.1070 (2) | 0.63278 (14) | 0.2448 (4) | 0.0658 (8) | |
| H1A | 0.1561 | 0.6116 | 0.2870 | 0.099* | |
| H1B | 0.0970 | 0.6629 | 0.3201 | 0.099* | |
| H1C | 0.1141 | 0.6502 | 0.1428 | 0.099* | |
| O2 | 0.1721 (2) | 0.73912 (12) | 0.7426 (5) | 0.1005 (11) | |
| O5 | 0.3253 (3) | 0.66497 (17) | 0.3658 (4) | 0.1196 (14) | |
| O8 | 0.4341 (2) | 0.61129 (16) | 0.2440 (6) | 0.1222 (14) | |
| O6 | 0.2856 (2) | 0.59355 (16) | 0.1434 (5) | 0.1159 (12) | |
| O4 | 0.0539 (2) | 0.6843 (2) | 0.5835 (5) | 0.1218 (13) | |

| | | | | |
|---|---|---|---|---|
| Cl1 | 0.12968 (6) | 0.68032 (4) | 0.71443 (11) | 0.0502 (2) |
| Cl2 | 0.35009 (6) | 0.63848 (4) | 0.21107 (12) | 0.0580 (3) |
| C1 | 0.0299 (2) | 0.58913 (16) | 0.2194 (4) | 0.0544 (9) |
| C3 | −0.0302 (3) | 0.48757 (18) | 0.2325 (5) | 0.0674 (11) |
| H3 | −0.0236 | 0.4448 | 0.2587 | 0.081* |
| C2 | 0.0446 (3) | 0.52653 (16) | 0.2615 (4) | 0.0562 (9) |
| C4 | −0.1126 (3) | 0.5099 (2) | 0.1672 (5) | 0.0786 (12) |
| H4 | −0.1612 | 0.4825 | 0.1497 | 0.094* |
| C6 | −0.0529 (3) | 0.6145 (2) | 0.1543 (6) | 0.0713 (11) |
| C9 | 0.1350 (3) | 0.50228 (19) | 0.3346 (5) | 0.0723 (11) |
| H9A | 0.1554 | 0.5253 | 0.4414 | 0.087* |
| H9B | 0.1758 | 0.5118 | 0.2518 | 0.087* |
| C10 | 0.1413 (3) | 0.43215 (19) | 0.3754 (6) | 0.0896 (14) |
| H10A | 0.2019 | 0.4216 | 0.4210 | 0.134* |
| H10B | 0.1231 | 0.4085 | 0.2703 | 0.134* |
| H10C | 0.1030 | 0.4221 | 0.4606 | 0.134* |
| C5 | −0.1239 (3) | 0.5723 (2) | 0.1274 (6) | 0.0850 (13) |
| H5 | −0.1804 | 0.5869 | 0.0813 | 0.102* |
| C11 | 0.4615 (2) | 0.64544 (17) | 0.7574 (4) | 0.0548 (9) |
| C16 | 0.5406 (3) | 0.6799 (2) | 0.7952 (5) | 0.0634 (10) |
| C12 | 0.4577 (3) | 0.58089 (18) | 0.7432 (5) | 0.0647 (10) |
| C20 | 0.3702 (4) | 0.4774 (3) | 0.7116 (8) | 0.125 (2) |
| H20A | 0.3098 | 0.4624 | 0.6854 | 0.187* |
| H20B | 0.3956 | 0.4630 | 0.8258 | 0.187* |
| H20C | 0.4049 | 0.4616 | 0.6260 | 0.187* |
| C13 | 0.5396 (3) | 0.5487 (2) | 0.7667 (6) | 0.0885 (14) |
| H13 | 0.5405 | 0.5049 | 0.7598 | 0.106* |
| C19 | 0.3704 (3) | 0.5457 (2) | 0.7082 (7) | 0.0876 (14) |
| H19A | 0.3333 | 0.5600 | 0.7930 | 0.105* |
| H19B | 0.3411 | 0.5587 | 0.5938 | 0.105* |
| C17 | 0.5385 (3) | 0.7511 (2) | 0.8118 (6) | 0.0765 (11) |
| H17A | 0.5014 | 0.7616 | 0.9005 | 0.092* |
| H17B | 0.5093 | 0.7682 | 0.7017 | 0.092* |
| C15 | 0.6199 (3) | 0.6456 (3) | 0.8166 (6) | 0.0883 (14) |
| H15 | 0.6745 | 0.6663 | 0.8425 | 0.106* |
| C14 | 0.6174 (3) | 0.5811 (3) | 0.7994 (7) | 0.0945 (15) |
| H14 | 0.6710 | 0.5589 | 0.8106 | 0.113* |
| C18 | 0.6274 (4) | 0.7845 (3) | 0.8577 (8) | 0.120 (2) |
| H18A | 0.6176 | 0.8292 | 0.8628 | 0.180* |
| H18B | 0.6649 | 0.7755 | 0.7700 | 0.180* |
| H18C | 0.6562 | 0.7699 | 0.9694 | 0.180* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C7A | 0.044 (6) | 0.062 (9) | 0.168 (16) | 0.000 (6) | −0.007 (7) | 0.039 (9) |
| C7B | 0.119 (14) | 0.088 (8) | 0.130 (11) | −0.023 (9) | 0.083 (11) | −0.023 (8) |
| C8A | 0.050 (8) | 0.135 (6) | 0.187 (16) | 0.045 (7) | −0.002 (6) | 0.010 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8B | 0.050 (8) | 0.135 (6) | 0.187 (16) | 0.045 (7) | −0.002 (6) | 0.010 (9) |
| N2 | 0.0530 (17) | 0.0584 (16) | 0.0511 (16) | 0.0024 (14) | 0.0081 (14) | −0.0052 (13) |
| O3 | 0.146 (3) | 0.103 (2) | 0.0636 (17) | −0.043 (2) | 0.0447 (19) | −0.0017 (16) |
| O1 | 0.0757 (19) | 0.0764 (18) | 0.114 (2) | 0.0053 (15) | 0.0331 (18) | −0.0259 (16) |
| O7 | 0.107 (2) | 0.0746 (17) | 0.0590 (15) | −0.0023 (16) | 0.0237 (16) | 0.0112 (13) |
| N1 | 0.077 (2) | 0.0641 (19) | 0.0620 (18) | −0.0194 (16) | 0.0293 (16) | −0.0043 (14) |
| O2 | 0.128 (3) | 0.0523 (16) | 0.129 (3) | −0.0247 (17) | 0.044 (2) | −0.0107 (16) |
| O5 | 0.181 (4) | 0.136 (3) | 0.0490 (16) | 0.062 (3) | 0.041 (2) | 0.0098 (17) |
| O8 | 0.073 (2) | 0.082 (2) | 0.216 (4) | 0.0294 (18) | 0.034 (2) | 0.020 (2) |
| O6 | 0.108 (3) | 0.091 (2) | 0.149 (3) | −0.036 (2) | 0.022 (2) | −0.002 (2) |
| O4 | 0.083 (2) | 0.165 (3) | 0.107 (3) | 0.030 (2) | −0.020 (2) | −0.002 (2) |
| Cl1 | 0.0526 (5) | 0.0464 (4) | 0.0535 (5) | −0.0003 (4) | 0.0142 (4) | −0.0012 (4) |
| Cl2 | 0.0594 (6) | 0.0571 (5) | 0.0608 (5) | 0.0063 (5) | 0.0198 (4) | 0.0041 (4) |
| C1 | 0.062 (2) | 0.057 (2) | 0.0494 (18) | −0.0151 (18) | 0.0236 (17) | −0.0073 (15) |
| C3 | 0.086 (3) | 0.056 (2) | 0.064 (2) | −0.022 (2) | 0.021 (2) | −0.0045 (17) |
| C2 | 0.072 (3) | 0.055 (2) | 0.0449 (18) | −0.0150 (18) | 0.0206 (18) | −0.0058 (15) |
| C4 | 0.076 (3) | 0.091 (3) | 0.071 (3) | −0.033 (3) | 0.017 (2) | 0.001 (2) |
| C6 | 0.076 (3) | 0.068 (3) | 0.078 (3) | −0.004 (2) | 0.036 (2) | 0.009 (2) |
| C9 | 0.076 (3) | 0.072 (3) | 0.071 (3) | −0.010 (2) | 0.017 (2) | 0.004 (2) |
| C10 | 0.104 (4) | 0.075 (3) | 0.089 (3) | 0.004 (3) | 0.012 (3) | 0.012 (2) |
| C5 | 0.065 (3) | 0.111 (4) | 0.082 (3) | −0.006 (3) | 0.020 (2) | 0.013 (3) |
| C11 | 0.060 (2) | 0.067 (2) | 0.0380 (17) | 0.0130 (19) | 0.0068 (16) | −0.0003 (15) |
| C16 | 0.058 (2) | 0.079 (3) | 0.053 (2) | 0.007 (2) | 0.0057 (18) | −0.0018 (18) |
| C12 | 0.068 (3) | 0.065 (2) | 0.062 (2) | 0.009 (2) | 0.009 (2) | 0.0027 (18) |
| C20 | 0.101 (4) | 0.105 (4) | 0.163 (6) | 0.001 (3) | −0.001 (4) | 0.017 (4) |
| C13 | 0.082 (3) | 0.075 (3) | 0.110 (4) | 0.025 (3) | 0.020 (3) | 0.007 (3) |
| C19 | 0.077 (3) | 0.063 (3) | 0.121 (4) | 0.010 (2) | 0.006 (3) | −0.002 (2) |
| C17 | 0.060 (3) | 0.090 (3) | 0.078 (3) | −0.007 (2) | 0.007 (2) | −0.013 (2) |
| C15 | 0.057 (3) | 0.114 (4) | 0.091 (3) | 0.009 (3) | −0.001 (2) | 0.004 (3) |
| C14 | 0.066 (3) | 0.100 (4) | 0.119 (4) | 0.024 (3) | 0.017 (3) | 0.009 (3) |
| C18 | 0.095 (4) | 0.118 (4) | 0.145 (5) | −0.031 (3) | 0.010 (4) | −0.024 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C7A—C8A | 1.22 (3) | C2—C9 | 1.493 (5) |
| C7A—C6 | 1.599 (17) | C4—C5 | 1.363 (6) |
| C7A—H7A1 | 0.9700 | C4—H4 | 0.9300 |
| C7A—H7A2 | 0.9700 | C6—C5 | 1.390 (6) |
| C7B—C8B | 1.38 (2) | C9—C10 | 1.520 (5) |
| C7B—C6 | 1.468 (19) | C9—H9A | 0.9700 |
| C7B—H7B1 | 0.9700 | C9—H9B | 0.9700 |
| C7B—H7B2 | 0.9700 | C10—H10A | 0.9600 |
| C8A—H8A1 | 0.9600 | C10—H10B | 0.9600 |
| C8A—H8A2 | 0.9600 | C10—H10C | 0.9600 |
| C8A—H8A3 | 0.9600 | C5—H5 | 0.9300 |
| C8B—H8B1 | 0.9600 | C11—C12 | 1.373 (5) |
| C8B—H8B2 | 0.9600 | C11—C16 | 1.395 (5) |
| C8B—H8B3 | 0.9600 | C16—C15 | 1.392 (6) |

| | | | |
|---|---|---|---|
| N2—C11 | 1.471 (4) | C16—C17 | 1.515 (6) |
| N2—H2A | 0.8900 | C12—C13 | 1.402 (5) |
| N2—H2B | 0.8900 | C12—C19 | 1.506 (6) |
| N2—H2C | 0.8900 | C20—C19 | 1.446 (6) |
| O3—Cl1 | 1.404 (3) | C20—H20A | 0.9600 |
| O1—Cl1 | 1.416 (3) | C20—H20B | 0.9600 |
| O7—Cl2 | 1.443 (3) | C20—H20C | 0.9600 |
| N1—C1 | 1.478 (4) | C13—C14 | 1.354 (6) |
| N1—H1A | 0.8900 | C13—H13 | 0.9300 |
| N1—H1B | 0.8900 | C19—H19A | 0.9700 |
| N1—H1C | 0.8900 | C19—H19B | 0.9700 |
| O2—Cl1 | 1.404 (3) | C17—C18 | 1.515 (6) |
| O5—Cl2 | 1.417 (3) | C17—H17A | 0.9700 |
| O8—Cl2 | 1.385 (3) | C17—H17B | 0.9700 |
| O6—Cl2 | 1.409 (3) | C15—C14 | 1.374 (6) |
| O4—Cl1 | 1.418 (3) | C15—H15 | 0.9300 |
| C1—C2 | 1.376 (5) | C14—H14 | 0.9300 |
| C1—C6 | 1.389 (5) | C18—H18A | 0.9600 |
| C3—C4 | 1.360 (6) | C18—H18B | 0.9600 |
| C3—C2 | 1.391 (5) | C18—H18C | 0.9600 |
| C3—H3 | 0.9300 | | |

| | | | |
|---|---|---|---|
| C8A—C7A—C6 | 115.0 (16) | C5—C6—C7B | 110.6 (8) |
| C8A—C7A—H7A1 | 108.5 | C1—C6—C7A | 114.6 (6) |
| C6—C7A—H7A1 | 108.5 | C5—C6—C7A | 128.9 (7) |
| C8A—C7A—H7A2 | 108.5 | C7B—C6—C7A | 21.4 (10) |
| C6—C7A—H7A2 | 108.5 | C2—C9—C10 | 116.3 (4) |
| H7A1—C7A—H7A2 | 107.5 | C2—C9—H9A | 108.2 |
| C8B—C7B—C6 | 134.4 (14) | C10—C9—H9A | 108.2 |
| C8B—C7B—H7B1 | 103.6 | C2—C9—H9B | 108.2 |
| C6—C7B—H7B1 | 103.6 | C10—C9—H9B | 108.2 |
| C8B—C7B—H7B2 | 103.6 | H9A—C9—H9B | 107.4 |
| C6—C7B—H7B2 | 103.6 | C9—C10—H10A | 109.5 |
| H7B1—C7B—H7B2 | 105.3 | C9—C10—H10B | 109.5 |
| C7A—C8A—H8A1 | 109.5 | H10A—C10—H10B | 109.5 |
| C7A—C8A—H8A2 | 109.5 | C9—C10—H10C | 109.5 |
| H8A1—C8A—H8A2 | 109.5 | H10A—C10—H10C | 109.5 |
| C7A—C8A—H8A3 | 109.5 | H10B—C10—H10C | 109.5 |
| H8A1—C8A—H8A3 | 109.5 | C4—C5—C6 | 121.4 (5) |
| H8A2—C8A—H8A3 | 109.5 | C4—C5—H5 | 119.3 |
| C7B—C8B—H8B1 | 109.5 | C6—C5—H5 | 119.3 |
| C7B—C8B—H8B2 | 109.5 | C12—C11—C16 | 124.3 (4) |
| H8B1—C8B—H8B2 | 109.5 | C12—C11—N2 | 117.6 (3) |
| C7B—C8B—H8B3 | 109.5 | C16—C11—N2 | 118.1 (3) |
| H8B1—C8B—H8B3 | 109.5 | C15—C16—C11 | 116.7 (4) |
| H8B2—C8B—H8B3 | 109.5 | C15—C16—C17 | 122.5 (4) |
| C11—N2—H2A | 109.5 | C11—C16—C17 | 120.8 (3) |
| C11—N2—H2B | 109.5 | C11—C12—C13 | 116.7 (4) |

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|---|---|---|---|
| H2A—N2—H2B | 109.5 | C11—C12—C19 | 122.3 (4) |
| C11—N2—H2C | 109.5 | C13—C12—C19 | 121.0 (4) |
| H2A—N2—H2C | 109.5 | C19—C20—H20A | 109.5 |
| H2B—N2—H2C | 109.5 | C19—C20—H20B | 109.5 |
| C1—N1—H1A | 109.5 | H20A—C20—H20B | 109.5 |
| C1—N1—H1B | 109.5 | C19—C20—H20C | 109.5 |
| H1A—N1—H1B | 109.5 | H20A—C20—H20C | 109.5 |
| C1—N1—H1C | 109.5 | H20B—C20—H20C | 109.5 |
| H1A—N1—H1C | 109.5 | C14—C13—C12 | 120.3 (4) |
| H1B—N1—H1C | 109.5 | C14—C13—H13 | 119.9 |
| O3—Cl1—O2 | 110.8 (2) | C12—C13—H13 | 119.9 |
| O3—Cl1—O1 | 110.4 (2) | C20—C19—C12 | 119.8 (4) |
| O2—Cl1—O1 | 109.79 (19) | C20—C19—H19A | 107.4 |
| O3—Cl1—O4 | 108.9 (2) | C12—C19—H19A | 107.4 |
| O2—Cl1—O4 | 111.2 (2) | C20—C19—H19B | 107.4 |
| O1—Cl1—O4 | 105.6 (2) | C12—C19—H19B | 107.4 |
| O8—Cl2—O6 | 110.8 (2) | H19A—C19—H19B | 106.9 |
| O8—Cl2—O5 | 110.8 (3) | C16—C17—C18 | 117.2 (4) |
| O6—Cl2—O5 | 109.2 (2) | C16—C17—H17A | 108.0 |
| O8—Cl2—O7 | 110.6 (2) | C18—C17—H17A | 108.0 |
| O6—Cl2—O7 | 109.1 (2) | C16—C17—H17B | 108.0 |
| O5—Cl2—O7 | 106.27 (19) | C18—C17—H17B | 108.0 |
| C2—C1—C6 | 124.4 (3) | H17A—C17—H17B | 107.2 |
| C2—C1—N1 | 118.4 (3) | C14—C15—C16 | 119.7 (5) |
| C6—C1—N1 | 117.3 (3) | C14—C15—H15 | 120.1 |
| C4—C3—C2 | 122.2 (4) | C16—C15—H15 | 120.1 |
| C4—C3—H3 | 118.9 | C13—C14—C15 | 122.3 (5) |
| C2—C3—H3 | 118.9 | C13—C14—H14 | 118.8 |
| C1—C2—C3 | 115.8 (4) | C15—C14—H14 | 118.8 |
| C1—C2—C9 | 121.9 (3) | C17—C18—H18A | 109.5 |
| C3—C2—C9 | 122.3 (3) | C17—C18—H18B | 109.5 |
| C3—C4—C5 | 120.0 (4) | H18A—C18—H18B | 109.5 |
| C3—C4—H4 | 120.0 | C17—C18—H18C | 109.5 |
| C5—C4—H4 | 120.0 | H18A—C18—H18C | 109.5 |
| C1—C6—C5 | 116.2 (4) | H18B—C18—H18C | 109.5 |
| C1—C6—C7B | 132.9 (8) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O6 | 0.89 | 2.41 | 3.029 (4) | 127 |
| N1—H1*B*···O4 | 0.89 | 2.27 | 3.043 (5) | 146 |
| N1—H1*B*···O2$^{i}$ | 0.89 | 2.48 | 2.889 (4) | 109 |
| N1—H1*C*···O3$^{ii}$ | 0.89 | 2.10 | 2.935 (4) | 157 |
| N1—H1*C*···O2$^{i}$ | 0.89 | 2.58 | 2.889 (4) | 101 |
| N2—H2*A*···O1 | 0.89 | 2.17 | 2.971 (4) | 149 |
| N2—H2*B*···O5 | 0.89 | 2.39 | 2.875 (4) | 114 |
| N2—H2*B*···O7$^{iii}$ | 0.89 | 2.24 | 2.991 (4) | 141 |

| | | | | |
|---|---|---|---|---|
| N2—H2*C*···O7$^{iv}$ | 0.89 | 2.03 | 2.805 (3) | 144 |
| C3—H3···O3$^{v}$ | 0.93 | 2.60 | 3.321 (5) | 134 |
| C13—H13···O8$^{vi}$ | 0.93 | 2.49 | 3.418 (6) | 172 |

Symmetry codes: (i) $x$, $-y+3/2$, $z-1/2$; (ii) $x$, $y$, $z-1$; (iii) $x$, $-y+3/2$, $z+1/2$; (iv) $x$, $y$, $z+1$; (v) $-x$, $-y+1$, $-z+1$; (vi) $-x+1$, $-y+1$, $-z+1$.